Vol 5 Issue 4 May 2015

ISSN No : 2230-7850

International Multidisciplinary Research Journal

Indian Streams Research Journal

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RNI MAHMUL/2011/38595 **ISSN No.2230-7850**

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International Recognized Double-Blind Peer Reviewed Multidisciplinary Research Journal
Indian Streams Research Journal
ISSN 2230-7850 Impact Factor : 3.1560(UIF)
Volume - 5 | Issue - 4 | May - 2015 Available online at www.isrj.org

LIQUID LIQUID EXTRACTION OF PLATINUM (IV) WITH CYANEX 301

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Short Profile

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ABSTRACT:

This paper describes experimental work of solvent extraction of Pt (IV) from acidic aqueous media with Cyanex 301 as extractant in chloroform. Various parameters such as acid concentration, reagent concentration, $SnCl_2$ concentration, shaking time, effect of diluents and effect of foreign ions were studied. Quantitative extraction of Pt (IV) in 0.6M HCl and 1.1 ml of 1M $SnCl_2$ stannous chloride was possible with 0.08 M Cyanex 301 after 75 seconds of shaking .The yellow coloured Pt-Cyanex 301 complex exhibits maximum absorption 425nm where extraction of reagent was found to be negligible. The complex is stable more than 48 hrs. The probable composition of the species has been deduced from the extraction data. Beer-Lambert law is obeyed in the concentration range 30µg -160µg with Sandell's sensitivity of 3.824×10^{-3} µg/mL/cm^2 and molar absorptivity of 51,411 mole^{-1}.cm^{-1}.dm^3. A study of effect of diverse ions on the extraction showed that several metals ions like Fe^{+2}, Fe^{+3}, Mn^{+2}, Mg^{+2}, Ti^{+4}, Sr^{+2} do not interfere during the extraction and estimation of platinum. The data have been successfully employed for the separation of binary mixtures containing the non interfering metal ions. The optimized conditions of separation have been successfully utilized to recover metal from real samples.

KEYWORDS
Extraction, Platinum, Cyanex 301, Chloroform.

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INTRODUCTION :

Platinum is one of the noble metal and a lustrous, ductile, and malleable, silver-white metal.[1]. The most common oxidation states of platinum are +2 and +4. Although elemental platinum is generally unreactive, it dissolves in hot *aqua regia* to give aqueouschloroplatinic acid (H_2PtCl_6):[2]Platinum is the least reactive metal and due to its corrosion resistance, it is not as toxic as some other metals.[3] Compounds containing platinum, such s- cisplatin, oxaliplatin and carboplatin are applied in chemotherapy against certain types of cancer.[4] By itself, it has various applications in photography, zinc etchings, indelible ink, plating, mirrors, porcelain coloring, and as a catalyst.[5] Platinum (IV) oxide, PtO_2, also known as Adams' catalyst, is a black powder that is soluble in KOH solutions and concentrated acids.[6] PtO_2 and the less common PtO both decompose upon heating.[1] Platinum also strongly catalyzes the decomposition of hydrogen peroxide into water and oxygen.[7] Platinum-cobalt, an alloy of roughly three parts platinum and one part cobalt, is used to make relatively strong permanent magnets.[5] Platinum-based anodes are used in ships, pipelines, and steel piers.[8]

Reference reveals that several reagents have been used for the spectrophotometric determination of platinum .[9-25] Platinum is also extractable from the acidic medium with various reagents. [26-40] It has also been extracted in the presence of 3,4-Diaminobenzoic acid[41], 5-phenyl,4-methyl-5-phenyl 1,2-thiole-3-thione [42] benzoin α-oxime [43] , benzyl a-onoxime44, thiobenzohydrazide[45], mezapine hydrochloride [46], 2,2'-Diamino diplenyl disulphide [47], N-(2-Mercapto phenyl) salicyladimine[48], 2-pyridyl-2-thienylß-ketoxime [49], 1,5-Diphenyl dithio biuret [50].The most frequently studied solvent for the extaction of platinum from chloride medium is chlororform [51-55], 1,2,Dichlororethane [56-57], carbon tetrachloride[58-61].

From above literature survey, it is obvious that existing methods for the determination of platinum have their own limitations[17, 18, 25, 34, 35, 37, 40, 43, 44] .The proposed method is simple , rapid for the separation and spectrophotometric determination of Platinum using Cyanex 301. Cyanex-301 [bis (2, 4, 4 trimethylpentyl) dithiophosphonic acid] marketed by Cytec Inc. Canada has been used as an extractant for some metal ions [62-74].

2. MATERIALS AND METHODS:

2.1 Stock Solution

All the chemicals (E. Merck) and diluents used in the present experimental studies were of Analytical Reagent grade. The extractant Cyanex 301 was supplied by Cytec Inc. Canada was used without further purification.

Stock solutions of various cations, anions were prepared from their respective salts. (Table .3) by taking proper precautions.

Pt (IV) stock solution was prepared by dissolving 0.174g of Platinum tetrachloride in a 100 mL std.measuring flask with distilled water containing about 5 mL conc. HCl.

2.2 Standardization of Platinum by gravimetric method:

Platinum was standardized gravimetrically as metallic platinum using formic acid as reducing

agent3. 3.0 g of anhydrous sodium acetate (pH at 5-6) and 1.0 mL of formic acid were added to the chloroplatinic acid solution containing about 5 mg of platinum in 25 mL solution and digested on water bath, cooled and filtered it through sintered glass crucible. Thereafter it was washed with distilled water till free from chloride, dried and ignited the filter paper in contact with the precipitate to constant weight. Weighed it as metallic platinum.

All absorbance measurements were carried out on 'Spectronic Genesis 8'UV- Visible spectrophotometer using 10mm path length quartz cuvettes.

2.3 General Extraction Procedure:

To an aliquot of the aqueous solution containing Platinum (IV) was added 1.1 mL of 1M $SnCl_2$ to which was added concentrated hydrochloric acid to make it 0.6 M in a total volume of 15mL. The solution was transferred into a 125mL separating funnel and shaken for 75 sec. with 15 mL of 0.08M Cyanex 301 solution in chloroform. After allowing the two phases to separate, the organic phase was collected in a 25mL standard measuring flask and diluted up to the mark with chloroform. A small quantity of anhydrous Sodium Sulphate was added to all the 25mL flasks to absorb the moisture. The absorbance of the extract was measured between 200 to 400 nm against blank. A Pt (IV) - Cyanex 301 complex in organic phase exhibits maximum absorption at 425nm where absorption of reagent was found to be negligible. Hence the wavelength 425 nm was chosen for further studies.

3. RESULTS AND DISCUSSION:

3.1.1 Absorption Spectrum:

The absorption of Platinum- Cyanex complex was studied over a wavelength range of 200-500 nm. The golden yellow coloured complex exhibited absorption maxima at 425nm (Fig 1). At this wavelength the absorption of the reagent was negligible. Therefore, the wavelength of 425nm was chosen for all further measurements (fig.1)

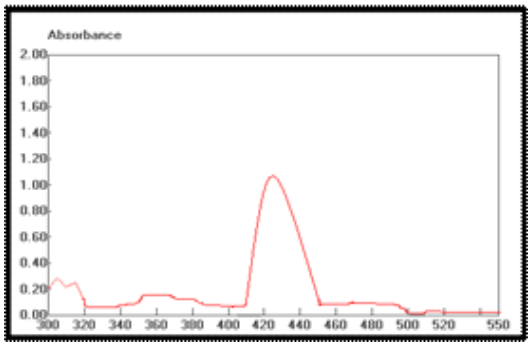


Fig.1. Absorption Spectra

3.1.2 Effect of reagent concentration:

The optimum concentration of Cyanex 301 for quantitative extraction of Pt (IV) was ascertained by extraction with varying concentrations of Cyanex 301 from 0.01-0.30 mol dm^{-3} in chloroform . The

extraction was quantitative with 0.08 mol dm^{-3} Cyanex 301 (Fig 2). Hence 15mL of 0.08 mol dm^{-3} Cyanex 301 in Chloroform was used throughout the study.

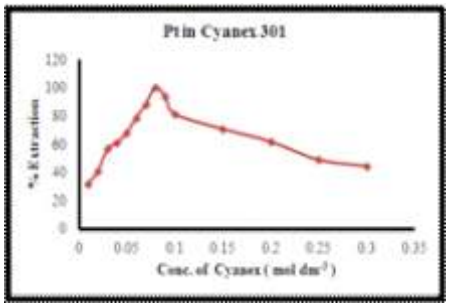


Fig.2. Cyanex Concentration

3.1.3 Effect of Hydrochloric acid concentration:

The effect of the molarity of hydrochloric acid concentration on the absorbance of the extract was studied using the recommended procedure. By varying concentration of HCl between 0.1 to 2.0 moldm^{-3}. The absorbance of extract was found to be maximum at 0.6 mol dm^{-3}. Thus 0.6 mol dm^{-3} HCl concentration was used for subsequent studies. (Fig.3)

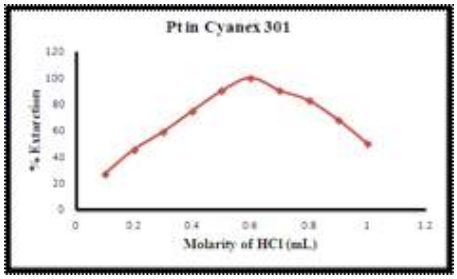


Fig.3. HCl Concentration

3.1.4 Effect of Stannous Chloride Concentration

Platinum was extracted in the presence of $SnCl_2$ which was used as a labializing agent. When tin is added to the solution, Platinum gets activated. This may be due to the reduction of Pt (IV) to Pt (II) with $SnCl_2$ and thus making the complex very easily extractable into Cyanex 301. In the absence of SnCl2, Platinum does not get extracted into Cyanex 301 using the recommended procedure. The effect of the molarity of $SnCl_2$ in the range of 0.033-0.100 mol dm^{-3} on the absorbance of the extract was studied using the recommended procedure at 425 nm with a sample containing 100µg of Platinum (IV) and 0.6 mol dm^{-3} HCl. The extraction was done using 15mL of 0.08 mol dm^{-3} Cyanex 301 in Chloroform. The absorbance was maximum for 0.0730 mol dm^{-3} of $SnCl_2$. Hence, all the extractions were carried out using 0.0730 mol dm^{-3} of $SnCl_2$. (Fig.4)

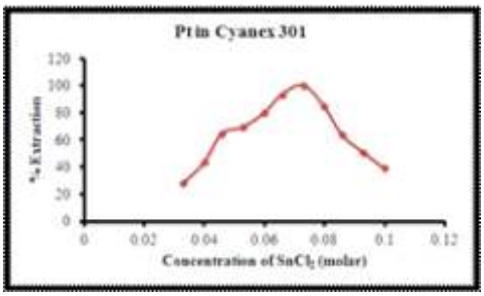


Fig.4. $SnCl_2$ Concentration

3.1.5 Effect of various Diluents:

To find out the effect of diluents on extraction, the reagent 0.08 mol dm^{-3} of Cyanex 301 was prepared in different diluents and was used for extraction from aqueous phase containing 100 µg of Platinum in 0.6 mol dm^{-3} HCl, 0.0730 mol dm^{-3} $SnCl_2$. For the diluents study the absorbance and % extraction of Platinum decreased in the order: Chloroform (99.99%), Toluene (95.47%), Carbon Tetrachloride (96.34%), Benzene (89.52%), Xylene (80.47%), Cyclohexane (65.48%). (Fig.5) Chloroform gave quantitative extraction and hence for all further studies chloroform was chosen as diluents.

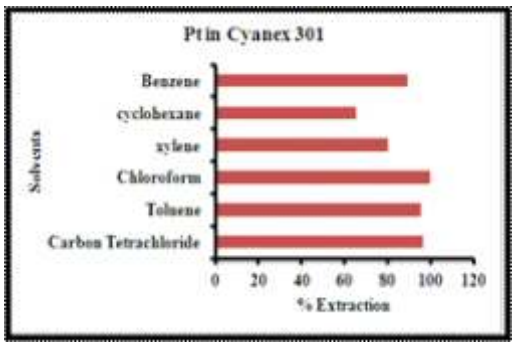


Fig.5. Effect of Diluents

3.1.6 Effect of Shaking period:

The absorbance of the extract obtained by shaking the aqueous phase containing 100µg Platinum (IV) + 1.1 mL of 1M $SnCl_2$ diluted to 15mL with 0.6 M Hydrochloric acid, with an organic phase containing 0.08 M Cyanex 301 in Chloroform was measured, for varying time periods from 15 sec to 120 sec. It was observed that the extraction was quantitative after 75 sec. of equilibration. Hence, optimum period chosen for shaking was 75 sec. (Fig.6)

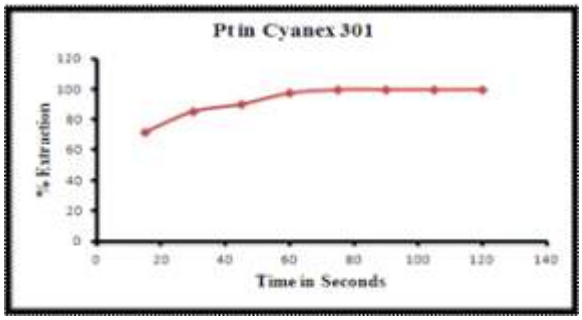


Fig. 6 Equilibration Period

4. Optimum Conditions for Extraction of Platinum (Table.1)

Parameter	Optimum Condition
Platinum(IV)	100 µg/mL
Hydrochloric acid concentration	0.6 mol dm^{-3}
$SnCl_2$ concentration	0.0730 mol dm^{-3}
Cyanex 301 concentration	0.08 mol dm^{-3}
Shaking Time	75 seconds
Diluent	Chloroform

5.1 Validity of Beer-Lambert law:

A calibration graph (Fig.7) for determination of Platinum was prepared under optimum experimental condition (0.0730 mol dm-3 $SnCl_2$, 0.6 mol dm^{-3} Hcl, and 0.08 mol dm^{-3} Cyanex in Chloroform). Beer's law was found to be obeyed in the range of 30 to 160 µg of Platinum at 425 nm. (Fig.7)

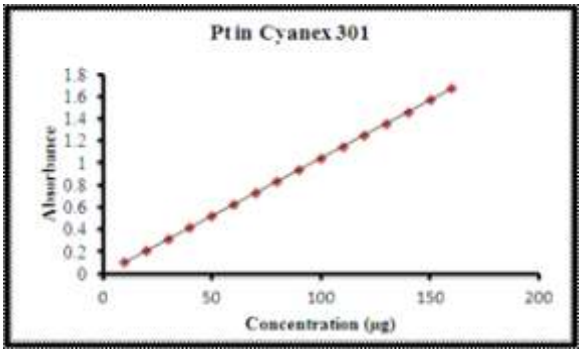


Fig.7. Beer's law plot

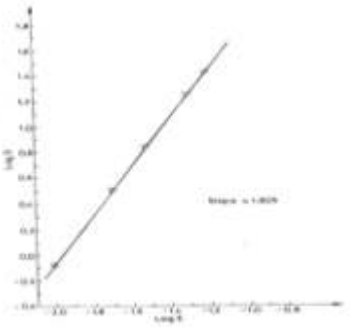
5.2. Spectrophotometric Data for the Determination of Platinum after Extraction with Cyanex 301 (Table.2)

The molar absorptivity of complex calculated was found to be 51,411 x 104 $mole^{-1}.cm^{-1}.dm^3$. At 425 nm Sandell's sensitivity calculated on the basis of total Platinum present is 3.824 $x10^{-3}$ µg cm^{-2} .The spectral characteristic are given in Table.2.

Molar absorptivity	51,411 x $10^4 mole^{-1}.cm^{-1}.dm^3$
Sandell's sensitivity	3.824 $x10^{-3}$ µg cm^{-2}
Mean absorbance of 6 determinations	1.046
Beer's law range	30-160mg/mL
Standard deviation	3.450 x 10^{-3}
Coefficient of variation	0.32 %

5.3 Nature of extracted species

An attempt was made to find out the probable composition of the extracted species from a plot of log D vs. log C (Cyanex 301) at fixed acid and $SnCl_2$ concentration. The slope of this plot was found to be 1.095 ≈2indicating the complex to be 1:2 with respect to Cyanex 301, thus confirming the oxidation state of Platinum as +2 in the extracted species. (Fig. 8)



7. EFFECT OF DIVERSE IONS & BINARY SEPARATION:

7.1 Effect of diverse ions

The extraction of Pt (IV) was carried out according to the recommended procedure to examine the effect of interference from various foreign ions. The tolerance limit was set at an amount to cause an error of + 2% in the recovery of the metal ion. It was observed that, a large number of cations and anions were tolerated (Table .3)

Table.3: Effect of foreign ions

Tolerance Ratio Pt(Iv): Diverse ions	Diverse Ions	
	Cation	Anions
Interference	Cu^{+2}, Pb^{+2}, Cd^{+2}, Co^{+2}, Pd^{+2}, Rh^{+3}, Ru^{+3}, Hg^{+2}	-----
1:10	Cd^{+2}, Bi^{+3}, Mo^{+6}, Zr^{+4}, Ni^{+2}, Zn^{+2}, Th^{+4}	---------
1:15	Co^{+2}, La^{+3}, Al^{+3}, Ba^{+2}, Sb^{+3}	
1:20	Be^{+2}, Ce^{+4}, K^{+}	SCN^{-}, Oxalate
1:25	Na^{+}, Ca^{+2}	F^{-}, I^{-}, CO_2^{-3}, Br^{-}
1:30	Sr^{+2}, Ti^{+4}, Te^{+2}, Mg^{+2}, Fe^{+2}, Fe^{+3}, Mn^{+2}	SO_3^{2-}, SO_4^{2-}, NO_3^{-}

7.2 Binary Separations of Platinum (IV)

Ions such as as Sr^{+2}, Ti^{+4}, Te^{+2}, Mg^{+2}, Fe^{+2}, Fe^{+3}, Mn^{+2}, SO_3^{2-}, $NH4$, SO_4^{2-}, NO^{3-} do not get extracted into Cyanex 301 under optimum extraction conditions for Platinum(IV) up to a certain concentration. Hence, it was possible to separate them from their binary mixtures. The unextracted Ti^{+4}, Mg^{+2}, Fe^{+2}, Fe^{+3}, Mn^{+2} was determined spectrophotometrically by known methods (Table. 4)

Table.4 Binary Separation of Platinum

Composition µg	Recovery of Pt(IV)* %	Coefficient of Variation	Recovery of the added ion* %	Coefficient of Variation	Estimation procedure for the added ion and its reference
Pt(IV):Ti(IV) 100 : 100	99.99	0.39%	95.61	0.48%	Hydrogen peroxide Method [75]
Pt(IV):Ca(II) 100 : 100	99.99	0.67 %	97.25	0.72%	Murexide Method [76]
Pt(IV): Mn(II) 100 : 100	99.99	0.76 %	98.71	0.65%	Potassium Periodate Method [77]
Pt(IV): Fe(III) 100 : 100	99.99	0.53%	96.49	0.47%	1,10- Phenonthrolin Method [78]
Pt(IV): Fe(II) 100 : 100	99.99	44%	96.76	0.40%	1,10- Phenonthrolin Method [78]

* Mean of five determinations

8. ANALYSIS OF PLATINUM (IV) IN REAL SAMPLES:

8.1.4. Application to Recovery of Platinum from Pt-Charcoal.

Pt-Charcoal is a catalyst used in reduction reaction, nitro to amine and aromatic cyclic to saturated cyclic compound. It contains 5% of Platinum. To prepare 1000µg Working Stock, 1 gm of catalyst was weighed and diluted to 20ml with diluents (2 ml conc. HCl +18 ml Water). It was shaken vigorously and filtered through Whatman filter paper No. 41 to get clear transparent solution. From this working stock, 0.1 ml of solution was taken for further extraction. Platinum in the extracted solution was determined by the proposed method and the results were compared with the analysis done using ICP.

Samples	Amount of Pt(IV) certified	Amount of Pt(IV) Found with Standard method	Amount of Pt(IV) Found with Proposed method	% Found with Standard method	% Found with Proposed method	R.S.D. (%)
Pt-Charcoal	5%	4.99%	4.97%	99.99	98.25	0.71%

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